Docket No.: 1422-0705PUS1

(PATENT)

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of: Naoteru HONDA et al.

Application No.: 10/563,810

Filed: January 9, 2006

For: MINERAL COMPOSITION

Confirmation No.: 5357

Art Unit: 1615

Examiner: MERCIER, Melissa S.

# **DECLARATION UNDER 37 CFR 1.132**

COMMISSIONER FOR PATENTS P. O. Box 1450 Alexandria, VA 22313-1450

#### Madam:

- I, Tatsuo OKA, residing in Mie-ken, Japan, hereby declare and state as follows:
- I am a worker of Taiyo Kagaku Co., Ltd., who is assignee of U.S.
   Application Serial No. 10/563,810 filed on January 9, 2006, entitled MINERAL COMPOSITION. I am thoroughly familiar with the contents of said Application, its prosecution before the United States Patent and Trademark Office and the references cited therein.
- I am a graduate of Mic University, Faculty of Bioresources and received a master's degree in the year 1991, majoring in physical chemistry of marine foods.
- I have been employed in Taiyo Kagaku Co., Ltd. in the year 1991 and have been assigned to the Research Laboratories.

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 I have been involved in the research and development of mineral preparation for foods since 2000.

5. The following experiments were conducted by myself or under my direct supervision and control in order to verify the technical advantages of the present invention using a diglycerol fatty acid ester as an emulsifier over a triglycerol fatty acid ester and a pentaglycerol fatty acid ester described in the cited reference Hojo et al (U.S. Patent No. 7.264.834, hereinafter simply referred to as "Hojo").

### METHODS AND RESULTS

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In order to clarify the technical advantages of the mineral composition of the present invention, a comparative test was conducted between the inventive product in which a diglycerol fatty acid ester was used as an emulsifier and comparative products in which a triglycerol fatty acid ester or a pentaglycerol fatty acid ester was used as described in Hojo as detailed below.

# Sample I (Inventive Product)

One-thousand and nine-hundred grams of ion-exchanged water was heated to 60°C, and 20 g of diglycerol monolaurate (trade name: "SUNSOFT Q-12 D"; HLB = 9; monoester content: 80% by weight; specific gravity: 0.9; manufactured by Taiyo Kagaku Co., Ltd.) used in Example 1 of the present specification was mixed therewith while heating to dissolve. Eighty grams of ferric pyrophosphate (manufactured by Tomita Yakuhin Kogyo K. K.) was added thereto to prepare a liquid mixture. The liquid mixture was pulverized with DYNO-MILL "MULTI LAB" (manufactured by WAB), and a pulverized product was heated at 80°C for 30 minutes, and treated with a

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ultrasonic oscillator UD-200 (manufactured by TOMY SEIKO CO., LTD., graduated scale 8, 10 minutes), to give a dispersion having an average particle size of 0.25  $\mu$ m (Sample I).

On the other hand, Hojo describes in column 5, lines 21-22 that the polyglycerol fatty acid ester is more preferably "fatty acid esters such as triglycerol and pentaglycerol." Therefore, we shall use for comparative tests olcic acid ester, stearic acid ester, and lauric acid ester of triglycerol, and lauric acid ester, myristic acid ester, and stearic acid ester of pentaglycerol in the declaration.

## Sample II (Comparative Product I)

One-thousand and nine-hundred grams of ion-exchanged water was heated to 60°C, and 20 g of triglycerol mono-dioleate (trade name: "SUNSOFT A-171C"; HLB = 10.0; monoester content: 30% by weight; manufactured by Taiyo Kagaku Co., Ltd.) was mixed therewith while heating to dissolve. Eighty grams of ferric pyrophosphate (manufactured by Tomita Yakuhin Kogyo K. K.) was added thereto to prepare a liquid mixture. The liquid mixture was pulverized with DYNO-MILL "MULTI LAB" (manufactured by WAB), and a pulverized product was heated at 80°C for 30 minutes, and treated with a ultrasonic oscillator UD-200 (manufactured by TOMY SEIKO CO., LTD., graduated scale: 8, 10 minutes), to give a dispersion having an average particle size of 0.45 µm (Sample II).

# Sample III (Comparative Product II)

One-thousand and nine-hundred grams of ion-exchanged water was heated to 60°C, and 20 g of triglycerol mono-distearate (trade name: "SUNSOFT A-181C"; HLB

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= 10.0; monoester content: 30% by weight; manufactured by Taiyo Kagaku Co., Ltd.) was mixed therewith while heating to dissolve. Eighty grams of ferric pyrophosphate (manufactured by Tomita Yakuhin Kogyo K. K.) was added thereto to prepare a liquid mixture. The liquid mixture was pulverized with DYNO-MILL "MULTI LAB" (manufactured by WAB), and a pulverized product was heated at 80°C for 30 minutes, and treated with a ultrasonic oscillator UD-200 (manufactured by TOMY SEIKO CO., LTD., graduated scale: 8, 10 minutes), to give a dispersion having an average particle size of 0.41 µm (Sample III).

## Sample IV (Comparative Product III)

One-thousand and nine-hundred grams of ion-exchanged water was heated to 60°C, and 20 g of pentaglycerol trioleate (trade name: "SUNSOFT A-173E"; HLB = 7.0; monoester content: 5% by weight; manufactured by Taiyo Kagaku Co., Ltd.) was mixed therewith while heating to dissolve. Eighty grams of ferric pyrophosphate (manufactured by Tomita Yakuhin Kogyo K. K.) was added thereto to prepare a liquid mixture. The liquid mixture was pulverized with DYNO-MILL "MULTI LAB" (manufactured by WAB), and a pulverized product was heated at 80°C for 30 minutes, and treated with a ultrasonic oscillator UD-200 (manufactured by TOMY SEIKO CO., LTD., graduated scale: 8, 10 minutes), to give a dispersion having an average particle size of 0.46 um (Sample IV).

# Sample V (Comparative Product IV)

One-thousand and nine-hundred grams of ion-exchanged water was heated to 60°C, and 20 g of pentaglycerol trimyristate (trade name: "SUNSOFT A-143E"; HLB = 8.0; monoester content: 5% by weight; manufactured by Taiyo Kagaku Co., Ltd.) was

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mixed therewith while heating to dissolve. Eighty grams of ferric pyrophosphate (manufactured by Tomita Yakuhin Kogyo K. K.) was added thereto to prepare a liquid mixture. The liquid mixture was pulverized with DYNO-MILL "MULTI LAB" (manufactured by WAB), and a pulverized product was heated at 80°C for 30 minutes, and treated with a ultrasonic oscillator UD-200 (manufactured by TOMY SEIKO CO., LTD., graduated scale: 8, 10 minutes), to give a dispersion having an average particle size of 0.44 µm (Sample V).

## Sample VI (Comparative Product V)

One-thousand and nine-hundred grams of ion-exchanged water was heated to 60°C, and 20 g of pentaglycerol tristearate (trade name: "SUNSOFT A-183E"; HLB = 7.0; monoester content: 5% by weight; manufactured by Taiyo Kagaku Co., Ltd.) was mixed therewith while heating to dissolve. Eighty grams of ferric pyrophosphate (manufactured by Tomita Yakuhin Kogyo K. K.) was added thereto to prepare a liquid mixture. The liquid mixture was pulverized with DYNO-MILL "MULTI LAB" (manufactured by WAB), and a pulverized product was heated at 80°C for 30 minutes, and treated with a ultrasonic oscillator UD-200 (manufactured by TOMY SEIKO CO., LTD., graduated scale: 8, 10 minutes), to give a dispersion having an average particle size of 0.51 µm (Sample VI).

#### Sample VII (Comparative Product VI)

One-thousand and nine-hundred grams of ion-exchanged water was heated to 60°C, and 20 g of triglycerol monolaurate (trade name: "TRL-100"; HLB = 13.5; monoester content: 60% by weight; manufactured by Riken Vitamin Co., Ltd.) was mixed therewith while heating to dissolve. Eighty grams of ferric pyrophosphate

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(manufactured by Tomita Yakuhin Kogyo K. K.) was added thereto to prepare a liquid mixture. The liquid mixture was pulverized with DYNO-MILL "MULTI LAB" (manufactured by WAB), and a pulverized product was heated at 80°C for 30 minutes, and treated with a ultrasonic oscillator UD-200 (manufactured by TOMY SEIKO CO., LTD., graduated scale: 8, 10 minutes), to give a dispersion having an average particle size of 0.47 μm (Sample VII).

## Sample VIII (Comparative Product VII)

One-thousand and nine-hundred grams of ion-exchanged water was heated to 60°C, and 20 g of triglycerol monostearate (trade name: "TRS-100"; HLB = 11.8; monoester content: 60% by weight; manufactured by Riken Vitamin Co., Ltd.) was mixed therewith while heating to dissolve. Eighty grams of ferric pyrophosphate (manufactured by Tomita Yakuhin Kogyo K. K.) was added thereto to prepare a liquid mixture. The liquid mixture was pulverized with DYNO-MILL "MULTI LAB" (manufactured by WAB), and a pulverized product was heated at 80°C for 30 minutes, and treated with a ultrasonic oscillator UD-200 (manufactured by TOMY SEIKO CO., LTD., graduated scale: 8, 10 minutes), to give a dispersion having an average particle size of 0.55 µm (Sample VIII).

## Sample IX (Comparative Product VIII)

One-thousand and nine-hundred grams of ion-exchanged water was heated to 60°C, and 20 g of pentaglycerol monolaurate (trade name: "SUNSOFT A-121E"; HLB = 14; monoester content: 30% by weight; manufactured by Taiyo Kagaku Co., Ltd.) was mixed therewith while heating to dissolve. Eighty grams of ferric pyrophosphate (manufactured by Tomita Yakuhin Kogyo K. K.) was added thereto to prepare a liquid

mixture. The liquid mixture was pulverized with DYNO-MILL "MULTI LAB". (manufactured by WAB), and a pulverized product was heated at 80°C for 30 minutes, and treated with a ultrasonic oscillator UD-200 (manufactured by TOMY SEIKO CO., LTD., graduated scale: 8, 10 minutes), to give a dispersion having an average particle size of 0.26 µm (Sample IX).

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## Sample X (Comparative Product IX)

One-thousand and nine-hundred grams of ion-exchanged water was heated to 60°C, and 20 g of pentaglycerol monomyristate (trade name: "SUNSOFT A-141E"; HLB = 13; monoester content: 30% by weight; manufactured by Taiyo Kagaku Co., Ltd.) was mixed therewith while heating to dissolve. Eighty grams of ferric pyrophosphate (manufactured by Tomita Yakuhin Kogyo K. K.) was added thereto to prepare a liquid mixture. The liquid mixture was pulverized with DYNO-MILL "MULTI LAB" (manufactured by WAB), a pulverized product was heated at 80°C for 30 minutes, and treated with a ultrasonic oscillator UD-200 (manufactured by TOMY SEIKO CO., LTD., graduated scale: 8, 10 minutes), to give a dispersion having an average particle size of 0.24 um (Sample X).

# Sample XI (Comparative Product X)

One-thousand and nine-hundred grams of ion-exchanged water was heated to 60°C, and 20 g of pentaglycerol monostearate (trade name: "SUNSOFT A-181E"; HLB = 13; monoester content: 30% by weight; manufactured by Taiyo Kagaku Co., Ltd.) was mixed therewith while heating to dissolve. Eighty grams of ferric pyrophosphute (manufactured by Tomita Yakuhin Kogyo K. K.) was added thereto to prepare a liquid mixture. The liquid mixture was pulverized with DYNO-MILL "MULTI LAB"

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(manufactured by WAB), a pulverized product was heated at 80°C for 30 minutes, and treated with a ultrasonic oscillator UD-200 (manufactured by TOMY SEIKO CO., LTD., graduated scale: 8, 10 minutes), to give a dispersion having an average particle size of 0.25 µm (Sample XI).

## Evaluation Method

Inventive Product I and Comparative Products I to X were evaluated on storage ability in the same manner as in Test Example 2 of the present specification as detailed below.

Each of Inventive Product I and Comparative Products I to X was allowed to stand at 5°C, at 25°C, or at 40°C. The extent of occurrence of precipitation of the constituents in the dispersion was visually observed from the time immediately after the preparation to after 2 weeks. The results are shown in Table I.

The evaluations were made in accordance with the following criteria:

## Evaluation Criteria

No precipitates:	-	
Hardly any precipitates:	±	
Slight amount of precipitates:	+	
Some precipitates:	++	
Outstanding amounts of precipitates:	+++	

Table I

	Sample I	Sample II	Sample III	Sample IV	Sample V	Sample VI
5°C	_	±	±	+	+	+
25°C	_	. +	+	+	+	+
40°C	±	++	++	++-	++	++

- continued -

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	Sample I	Sample VII	Sample VIII	Sample IX	Sample X	Sample XI
5°C		+	+	++	++	+-+-
25°C		++	++	++	4-4-	++
40°C	±	+++	+-+-+		+	+

# DISCUSSION

It is clear from the results shown in Table I that the inventive product is found to suppress the occurrence of precipitation. The diglycerol fatty acid ester used in the present invention has evidently a more excellent effect of suppressing precipitates in the dispersion than the triglycerol or pentaglycerol fatty acid ester of Hojo. The reasons why such advantageous effects are obtained in the inventive product over the comparative products are considered to be as follows. The diglycerol fatty acid ester in the present invention has a monoester content as high as 50% by weight or more, and ester compounds other than the monoester, i.e. a diester, a triester, or the like, which are considered to function so as to inhibit interfacial adsorption to an insoluble metal salt are contained in smaller amounts, thereby making it more likely to allow the ester to adsorb to the insoluble metal salt. The diglycerol fatty acid ester in the present invention has an HLB of from 6 to 10. When the HLB of the diglycerol fatty acid ester

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is higher than 10, its affinity to water is stronger than that to the insoluble metal salt, thereby making it less likely to adsorb to the surface of an insoluble metal salt. If the HLB of triglycerol or pentaglycerol fatty acid ester is adjusted to 6 to 10, the monoester content is lowered than 50% by weight, and if the monomer content of the triglycerol or pentaglycerol fatty acid ester is adjusted to 50% by weight or more, its HLB becomes higher than 10, so that the effects as exhibited in the present invention are not obtained by the use of the triglycerol or pentaglycerol fatty acid ester.

Here, a test period is only taken at 2 weeks, an abusive test at 40°C is equivalent to a test at an ambient temperature for 3 months, showing prominent differences in effects.

# Statement Under 18 U.S.C. § 1001

The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

Tatsuo Oka

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Jan. 06, 2010

Date